[JP,10-189322,A]

Detailed Description of the Invention]
[0001]
[Industrial Application]This invention relates to a magnetic thin film, and relates to the soft magnetism thin film for the suitable magnetic head for use by high frequency, a thin film inductor, a thin film transformer, a thin film magnetic filter, etc. especially.
[0002]
[Description of the Prior Art]In recent years, high frequency-ization of various kinds of electron devices or a magnetic device is advanced with the miniaturization of electronic equipment. For example, in the magnetic head, the clock frequency is expected are made high to not less than hundreds of MHz from tens of present MHz in the near future. In addition, the correspondence to high-frequency-izing is searched for also about micro magnetic devices and noise filters, such as a thin film inductor and a transformer. However, the conventional magnetic metal thin films, such as a NiFe alloy, had the difficulty that it could not respond to such high frequency apparatus, for example, in order for eddy current loss to become large and for the amplitude permeability in high frequency to fall, since the specific resistance rho is low. There is also such a technological background and realization of the soft magnetism thin film which can respond to not less than hundreds of MHz high frequency is demanded.
[0003]It is the loss by an eddy current and magnetic resonance which generally becomes the cause of restricting the frequency characteristic of a magnetic material. If it sees about eddy current loss frequency first, it will decrease by thin-film-izing of a magnetic material, and high-specific-resistance-ization. About ferromagnetic resonant frequency, it is shifted more to the high frequency side enlarging saturation magnetization and by giving a large anisotropy field. That is, the magnetic material in which the characteristic outstanding in the high frequency region is shown constitutes high specific resistance and the thin film body of high saturation magnetic flux density.
[0004]By the way, the soft magnetism thin film with the granular structure which exists as a soft magnetism thin film with high specific resistance after the ferromagnetic metallic phase and the nonmagnetic insulator phase have carried out phase separation is already known. "Granular structure" here expresses the structure where a ferromagnetic metallic phase exists in the state where it distributed in the insulator phase as detailed particles.
[0005]For example, the soft magnetism thin film expressed with "Fe-M-O" (element more than a kind as which M is chosen from 3A group element and 4A group element) is indicated by JP,4-26105,A.
[0006]The soft magnetism thin film expressed with "Co-M-O" (it is chosen from 3A group element, 4A group element, and 5A group element, and M is an element more than a **** kind) is indicated by JP,4-62806,A.
[0007]The soft magnetism thin film with such granular structure is constituted from a ferromagnetic metallic element like Fe and Co, and this ferromagnetic metallic element by the element M which is easy to combine with oxygen, and oxygen.
[0008]According to the granular structure in the state where this ferromagnetic phase and a nonmagnetic oxide insulating phase carried out phase separation, high specific resistance is realizable. Namely, according to Institute of Electrical Engineers of Japan

MAGUNE textile study group data MAG-96-158. In the granular structure film of a "Co-M-O" (germanium, Sn, Si, aluminum as M) system, The membrane structure formed changes with heat of formation (namely, grade of the ease of carrying out of oxidation of M) of an M acid ghost, and the report of the purport that formation of a granular structure film is easy is made, so that a difference with the heat of formation of Co oxide is large M. Although the cause is not clarified about the granular structure film of a "Co-aluminum-O" system, it is also reported by by adding Pd that a big anisotropy field and good soft magnetism are acquired.

[0009]Now, although it is required for the high-specific-resistance-izing to make the rate of the insulator phase in a film increase in a soft magnetism thin film with the granular structure in which the ferromagnetic phase and the nonmagnetic oxide insulating phase carried out phase separation, Since an insulator phase was nonmagnetic, when the rate was made to increase, there was a difficulty that saturation magnetic flux density will decrease. That is, a raise in saturation density and high-specific-resistance-ization had the problem that an opposite relation consisted.

[0010]On the other hand, in JP,6-251939,A, the soft magnetism thin film which consists of two phases of alphaFe and Fe_4N as a thin film which has a ferromagnetic metallic phase and a ferromagnetic high-specific-resistance phase is indicated. Here, Fe_4N is a ferromagnetic with high saturation magnetic flux density, such as 1.6T, and since alphaFe forms 20 to 60% of whole products, the thin film of high saturation magnetic flux density, such as 1.78T-1.91T, is produced. however, since the specific resistance of said Fe_4N was not high, when the specific resistance as the whole thin film was a maximum of only about 100 microomegacm, it was obtained, and had a difficulty. Although the specific resistance of Fe_4N is not indicated, also when this occupies 60% of whole products, it is specifically conjectured to be 200-300 or less microomegacm, in view of a certain thing.

[0011]

[Problem(s) to be Solved by the Invention]This invention is made that SUBJECT found out in the conventional technology described above should be solved, and is a thing. While realizing the purpose and high specific resistance simultaneously and reducing the loss by an eddy current and ferromagnetic resonance, it is providing the magnetic thin film in which high magnetic permeability is shown also in high frequency.

[0012]

[Means for Solving the Problem]this invention person came to find out the following magnetic thin films, as a result of inquiring and inquiring wholeheartedly, in order to solve an aforementioned problem. Namely, a magnetic thin film, wherein mean particle diameter of metal particles from which a ferromagnetic insulating phase of 1000 or more microomegacm exists, and (1) ferromagnetism metallic phase and specific resistance constitute this ferromagnetic metallic phase is 20 nm or less.

[0013](2) A magnetic thin film of (1), wherein mean particle diameter of metal particles which constitute said ferromagnetic metallic phase is 10 nm or less.

[0014](3) A magnetic thin film of (1) or (2) being an oxide in which said ferromagnetic metallic phase uses at least one kind in iron and cobalt as main composing elements, and a ferromagnetic insulating phase uses iron and oxygen as main composing elements.

[0015](4) One magnetic thin film of (1) - (3) which contains at least one kind of a non-

magnetic metal element which does not form a solid solution substantially with said ferromagnetic metal, and cannot oxidize as easily as ferromagnetic metal at a rate below 20 atom %.
[0016](5) One magnetic thin film of (1) - (4), wherein non-magnetic metal elements which do not form a solid solution substantially with said ferromagnetic metal, and cannot oxidize as easily as ferromagnetic metal are Au, Ag, and Cu.
[0017]
[Function]According to this invention, the mean particle diameter of metal particles the ferromagnetic metallic phase and the ferromagnetic insulating phase of high specific resistance which are 20 nm or less by making it exist by granular structure. For example, also in not less than 500-MHz high frequency, the high amplitude permeability of 300 or more can be shown, and the soft magnetism thin film which has the high saturation magnetic flux density beyond 1.3T in addition to this can be provided. If this is said to details more, as an oxide which uses at least one kind in iron and cobalt as main composing elements about said ferromagnetic metallic phase, and uses iron and oxygen as main composing elements about a ferromagnetic insulating phase, The soft magnetism thin film concerning this invention can be obtained by making the non-magnetic metal element (for example, at least one kind of Au, Ag, and Cu) which does not form a solid solution substantially with said ferromagnetic metal, and cannot oxidize as easily as ferromagnetic metal contain at a rate below 20 atom %.
[0018]
[Embodiment of the Invention]The magnetic thin film concerning this invention has granular structure, and it is obtained by making it exist, where phase separation of the ferromagnetic predetermined metallic phase is carried out to the ferromagnetic insulator phase of correspondence. It is made for ********** of the metal particles which comprise that a ferromagnetic metallic phase and specific resistance make the ferromagnetic insulating phase of 1000 or more microomegacm exist [magnetic thin film / this] at details, and constitute this ferromagnetic metallic phase to be 20 nm or less more.
[0019]The magnetic thin film concerning this invention is constituted by making the ferromagnetic insulator phase which more specifically consists of a ferromagnetic metallic phase which used iron as the main ingredients, for example, and an iron oxide phase exist, where phase separation is carried out. namely, **** -- by distributing the ferromagnetic metallic phase which consists of detailed metal particles in a ferromagnetic insulating phase, in order to obtain soft magnetic characteristics, the thing of suitable granular structure is obtained. When such granular structure is used, a ferromagnetic metallic phase is made to dissociate by the ferromagnetic insulating phase, and specific resistance high as a result is shown. Since the insulating phase here also has ferromagnetism if it furthermore says, high saturation magnetic flux density will also be shown. In this way, in the magnetic thin film concerning this invention, while outstanding soft magnetic characteristics are shown, the conflicting high saturation magnetic flux density and high specific resistance are simultaneously realizable conventionally.
[0020]In addition to the iron illustrated previously, it can be used as metal which constitutes the aforementioned ferromagnetic metallic phase, being able to choose simple substance metal and these alloys of cobalt. The aforementioned iron, or simple substance metal and these alloys of cobalt can be used as main composing elements, and the

ferromagnetic metal it was made to contain other elements mentioned later can also be used. Here, it is preferred for the mean particle diameter of the metal particles which constitute said ferromagnetic metallic phase to use 20 nm or less (preferably 10 nm or less). When the mean particle diameter is larger than the above-mentioned value (namely, 20 nm), soft magnetic characteristics will deteriorate for the influence of crystal magnetic anisotropy. The value of mean particle diameter here points out the value obtained as a result of observation by the computed value and transmission electron microscope from the half breadth by an X diffraction.
[0021]The specific resistance is used as a material which constitutes the ferromagnetic insulator phase used in this invention, choosing the thing more than 1000microomegacm. When the specific resistance is lower than 1000microomegacm, the specific resistance of the whole thin film becomes low, and a high frequency characteristic will deteriorate as the result.
[0022]As a material which constitutes said ferromagnetic insulator phase, gammaFe_2O_3 which is an iron oxide, for example can be used. However, as a ferromagnetic insulator phase which constitutes the magnetic thin film concerning this invention, it may be a thing of the nonequilibrium state of a "Fe-O" system it was made to include locally the oxide phase of not only single-phase gammaFe_2O_3 but iron. The spinel ferrite of other ferromagnetic insulators, for example, Fe_3O_4, $ZnFe_2O_4$, and $MgFe_2O_4$, etc. can also constitute.
[0023]In the magnetic thin film concerning this invention, it is desirable to make at least one kind of the non-magnetic metal element which does not form a solid solution substantially with the ferromagnetic metal of correspondence, and cannot oxidize as easily as ferromagnetic metal contain at a rate below 20 atom %.

[0024]Here, "a solid solution is not substantially formed with ferromagnetic metal" means containing, when [else / when not forming a solid solution at all] there are few solid-solution limits as [below] 15 atom %. For example, when seen about Cu, to iron, the solid solution was formed within the limit of 1.8%, and the solid solution was formed within the limit of 10 to 12% to cobalt, but it expressed as mentioned above including such a case.
[0025]It is effective to add the non-magnetic metal element which does not form a solid solution substantially with the ferromagnetic metal of correspondence, and cannot oxidize as easily as ferromagnetic metal, in order to carry out minuteness making of the ferromagnetic metallic phase and to generate the suitable granular structure for realization of soft magnetic characteristics. The reason originates in the granular structure (namely, fine structure) of a nano-scale [soft magnetic characteristics / said].
It is because it has an effect which controls big and rough-ization of the crystal grain of a ferromagnetic metallic phase by the deposit to add said non-magnetic metal element. Since specific resistance is also reduced in addition to lowering saturation magnetic flux density by adding said non-magnetic metal element, it is preferred to make the addition below into 20 atom %. When it adds exceeding this limit, high saturation magnetic flux density is not only no longer obtained, but specific resistance will fall. Although it is also possible to choose the simple substance metallic element in two or more kinds of metallic elements made into an object as said non-magnetic metal element, two or more kinds of them may be chosen as a candidate for addition.

[0026]By not making a solid solution form substantially with the ferromagnetic metal which uses iron or cobalt as the main ingredients, in order to acquire minuteness making and the suitable granular structure for realization of soft magnetic characteristics for a ferromagnetic metallic phase, Au, Ag, and Cu can be mentioned as an effective non-magnetic metal element.
[0027]The soft magnetism thin film concerning this invention can be manufactured by applying a reactive sputtering method and a multicomponent target method, for example. In the former reactive sputtering method, ferromagnetic metal is targeted and it is manufactured by carrying out weld slag membrane formation in the atmosphere containing oxygen. Iron or the simple substance metal target of cobalt may be sufficient as the target at this time, and the multicomponent target combined with the alloy target containing one or more of them or said ferromagnetic metal may be sufficient as it. It may be a multicomponent target of the gestalt which combined said non-magnetic metal element further.
[0028]On the other hand by the latter multicomponent target method which is the gestalt which combined ferromagnetic metal and a ferromagnetic oxide. The multicomponent target which combined iron, the simple substance metal of cobalt or its alloy target, and ferromagnetic oxides, such as gammaFe_2O_3, The soft magnetism thin film concerning this invention is manufactured by carrying out weld slag membrane formation of the multicomponent target which combined the non-magnetic metal element furthermore described above in pure Ar atmosphere. Weld slag membrane formation here can also be performed in the mixed gas atmosphere containing oxygen.
[0029]As a sputtering technique applied to said weld slag membrane formation, the ion beam weld slag method, others, especially the thing restricted are not in addition to rf sputtering technique. Since the reaction at the time of the membrane formation is promoted, necessary heating can also be performed about the substrate which makes a soft magnetism thin film generate. When performing this substrate heating, since there is fear of oxygen diffusion, low-temperature heating at 200 ** or less is desirable.
[0030]As for the soft magnetism thin film concerning this invention, it is desirable to form membranes in a predetermined magnetic field for grant of the anisotropy field Hk. In addition, after forming membranes in no magnetic field, said Hk can also be given by heat-treating in a static magnetic field.
[0031]The soft magnetism thin film concerning this invention shows soft magnetic characteristics also in the state which has formed membranes. In order to improve to these soft magnetic characteristics, suitable heat treatment can also be performed. In this case, since oxygen in the ferromagnetic oxide phase which comprises an iron oxide may be spread in a ferromagnetic metallic phase for example, it is preferred to heat-treat at the temperature of 300 ** or less. When oxygen is spread by performing hot heat treatment exceeding this, granular structure will be destroyed and it stops showing good soft magnetic characteristics. The fall of saturation magnetic flux density and the fall of specific resistance may be produced by diffusion of this oxygen.
[0032]
[Example]Hereafter, the example and comparative example of this invention are shown, and this invention is explained further.
[0033]Sample production was performed using rf magnetron sputtering equipment.
[0034]The substrate was considered as indirect water cooling using the glass substrate

and glassy carbon board of the 0.5-mm-thick product number 7059 by Corning, Inc.
[0035]A target at the target of pure iron 100 mm in diameter, or pure cobalt. The gammaFe_2O_3 chip, the Fe-B (80:20at%) chip, and the non-magnetic metal chip of Au (gold) and V (vanadium) were used as the multicomponent target combined 0 to 15% by surface ratio as shown in "Table 1."
[0036]Here, Au in an example was added as non-magnetic metal which does not form a solid solution substantially to iron and cobalt.
[0037]V in a comparative example was added as non-magnetic metal which forms a solid solution to iron.
[0038]The specific resistance of the Fe-B (80:20at%) chip was 300microomegacm.
[0039]On the other hand, the specific resistance of the gammaFe_2O_3 chip was 5000 or more microomegacm, and the exact numerical value was incapable measurement.
[0040]Pure argon was used as sputtering ambient.
[0041]About some examples (examples 6 and 7), the sample was produced not using the chip of a ferromagnetic oxide by the reactive sputtering method using the mixed gas (argon + oxygen) as for which 2% of the partial pressure contained oxygen in sputtering ambient.
[0042]In order to give uniaxial magnetic anisotropy to a film sample, while forming the magnetic field of 250 Oe, it impressed with the permanent magnet in parallel with a substrates face. The membrane formation rate was about 2-40nm/second, and thickness set to about 100 nm for component analyses, and could be 1 micrometer the object for component analyses by EPMA mentioned later, specific resistance, and for evaluation of magnetic properties.
[0043]Said film sample was evaluated by the following measuring methods in the state which has formed membranes.
[0044](1) The component analysis was conducted by the Rutherford backscattering method (RBS) and an X-ray microanalyser (EPMA).
[0045](2) Measurement of the specific resistance rho was performed by the direct-current 4 terminal method.
[0046](3) The vibrating sample magnetometer (VSM) performed measurement of the saturation magnetic flux density Bs and the coercive force Hc.
[0047](4) About measurement of the amplitude permeability mu, it carried out in 500 MHz by the parallel line method.
[0048](5) With the application of Scherrer's formula, it asked for the crystal grain diameter D from the observation by a transmission electron microscope (TEM), and an X-ray diffraction method (XRD).
[0049]This evaluation result is as being shown in "Table 2." If it sees about Example 1, being indicated as "Fe-O8" expresses the composition ratio which makes O 8atom% and to which it sets the emainder to Fe, and it is [in / "Table 2"] the same also about other examples and a comparative example.
[0050]The effect of the above result to this invention is clear. That is, if Examples 1-9 in "Table 2" are contrasted with the comparative examples 10-16, the specific resistance rho in each example and the saturation magnetic flux density Bs are improved simultaneously, and the high amplitude permeability mu is obtained so that clearly.
[0051]According to the example of this invention, there are few losses also in high frequency and the magnetic thin film in which the high amplitude permeability mu is

obtained can be obtained.

[0052]

[Table 1]

試料番号		ターゲット	γFe2O3	Fe-B	非磁性金属	備考
1	実施例	純鉄	10	0	0	
2	実施例	純鉄	15	0	0	
3	実施例	純鉄	10	0	Au：0.5	
4	実施例	純鉄	10	0	Au：1.5	
5	実施例	純鉄	10	0	Au：10	
6	実施例	純鉄	0	0	0	反応性スパッタ
7	実施例	純鉄	0	0	Au：10	反応性スパッタ
8	実施例	純コバルト	10	0	0	
9	実施例	純コバルト	10	0	Au：1.5	
10	比較例	純鉄	0	0	0	
11	比較例	純鉄	10	0	Au：15	
12	比較例	純鉄	5	0	0	
13	比較例	純鉄	0	20	0	
14	比較例	純鉄	10	0	V：20	
15	比較例	純コバルト	0	0	0	
16	比較例	純コバルト	0	20	0	

（注）直径100mmのターゲット上のチップの面積比を%で表示した。

[0053]

[Table 2]

試料番号		膜組成 at%	Bs T	Hc Oe	ρ μΩcm	μ —	D nm
1	実施例	Fe−O8	1.7	1.9	750	550	9
2	実施例	Fe−O12	1.5	1.2	980	650	8
3	実施例	Fe−O8 −Au1	1.6	0.8	740	650	5
4	実施例	Fe−O8 −Au3	1.5	0.5	650	750	5
5	実施例	Fe−O8 −Au12	1.4	0.9	500	550	4
6	実施例	Fe−O10	1.6	1.5	600	520	7
7	実施例	Fe−O12−Au8	1.3	0.9	550	680	4
8	実施例	Co−Fe5−O8	1.4	1.1	680	600	8
9	実施例	Co−Fe5−O8−Au3	1.3	0.9	600	650	4
10	比較例	Fe	2.1	4.2	15	40	45
11	比較例	Fe−O8−Au22	0.8	26	280	30	4
12	比較例	Fe−O8	1.8	12	150	50	32
13	比較例	Fe−B5	1.6	5.4	80	70	25
14	比較例	Fe−O9−V23	0.9	32	290	50	31
15	比較例	Co	1.6	4.8	15	30	42
16	比較例	Co−Fe20−B5	1.7	3.2	65	120	27

[0054]